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IS 3026 (1991): Tin salts for electroplating [CHD 5 :  
Electroplating Chemicals and Photographic Materials]

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Bhartṛhari—Nītiśatakam

“Knowledge is such a treasure which cannot be stolen”





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भारतीय मानक  
विद्युत लेपन के लिए टिन लवण — विशिष्ट  
( दूसरा पुनरीक्षण )

*Indian Standard*  
**TIN SALTS FOR ELECTROPLATING —  
SPECIFICATION**  
*( Second Revision )*

UDC 661.881.27 : 621.357.7

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

## **FOREWORD**

This Indian Standard ( Second Revision ) was adopted by the Bureau of Indian Standards, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

This standard was originally published in 1964 covering only the sodium stannate salt for electroplating and was revised in 1968 with a view to specify the requirements for other tin salts also, such as stannous sulphate, potassium stannate and stannous chloride for electroplating to make it more comprehensive.

In this, the second revision of the standard, the requirements for sodium stannate and potassium stannate for electroplating industry have been modified and additional requirements for potassium stannate for use in electronics industry have been included. It is felt that the demand for the tin fluoroborate for electroplating is not much, and therefore, the inclusion of this salt in this standard has been postponed. It is expected that this revised standard will prove helpful to manufacturers in indigenous production of tin salts of desired quality.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

## Indian Standard

# TIN SALTS FOR ELECTROPLATING — SPECIFICATION

*( Second Revision )*

### **1 SCOPE**

**1.1** This standard prescribes the requirements and the methods of sampling and test for stannous sulphate, sodium stannate, potassium stannate and stannous chloride, used for electroplating.

### **2 REFERENCES**

The Indian Standards listed below are the necessary adjuncts to this standard:

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid ( <i>second revision</i> )
265 : 1987	Hydrochloric acid ( <i>third revision</i> )
266 : 1977	Sulphuric acid ( <i>second revision</i> )
323 : 1959	Rectified spirit ( <i>revised</i> )
1070 : 1977	Water for general laboratory use ( <i>second revision</i> )
2088 : 1983	Methods for determination of arsenic ( <i>second revision</i> )
2316 : 1968	Methods of preparation of standard solution for colorimetric and volumetric analysis ( <i>first revision</i> )
4161 : 1967	Nessler cylinders
9497 : 1980	Method for determination of sodium and potassium ( <i>flame photometric</i> )

### **3 REQUIREMENTS**

#### **3.1 Stannous Sulphate**

##### **3.1.1 Description**

The material shall be in the form of a pale cream coloured substance generally of a fine crystalline structure, free from dirt, foreign matter and visible impurities.

##### **3.1.2 Composition**

The material shall correspond in composition essentially to  $\text{SnSO}_4$ .

NOTE — Commercial stannous sulphate almost invariably contains some stannic sulphate. The minimum content of stannous tin specified automatically limits the maximum stannic sulphate content to a safe value.

**3.1.3** The material shall also comply with the requirements given in Table I, when tested according to the methods prescribed in Annex A.

#### **3.2 Sodium Stannate and Potassium Stannate**

##### **3.2.1 Description**

The material shall be in the form of colourless white crystals free from dirt, foreign matter and visible impurities.

**Table I Requirements for Stannous Sulphate**  
( Clause 3.1.3 )

<b>Sl No.</b>	<b>Characteristic</b>	<b>Requirement</b>	<b>Method of Test ( Ref to Cl No. of Annex A )</b>
<b>(1)</b>	<b>(2)</b>	<b>(3)</b>	<b>(4)</b>
i)	Insoluble matter, percent by mass, <i>Max</i>	0·5	A-2
ii)	Stannous tin (as Sn), percent by mass, <i>Min</i>	50·0*	A-3
iii)	Free sulphuric acid ( $\text{H}_2\text{SO}_4$ ), percent by mass, <i>Max</i>	2·0	A-4
iv)	Chloride (as Cl), percent by mass, <i>Max</i>	0·5	A-5
v)	Antimony (as Sb), percent by mass, <i>Max</i>	0·1	A-6
vi)	Arsenic (as As), percent by mass, <i>Max</i>	0·01	A-7

\*Equivalent to 90·4 percent by mass, *Min* of stannous sulphate.

**3.2.2 Composition**

Sodium stannate and potassium stannate shall correspond in composition essentially to  $\text{Na}_2\text{SnO}_3 \cdot 3\text{H}_2\text{O}$  and  $\text{K}_2\text{SnO}_3 \cdot 3\text{H}_2\text{O}$  respectively.

**3.2.3** The material shall also comply with the requirements given in Table 2 when tested according to the methods prescribed in Annex B.

**3.2.3.1** Potassium stannate to be used in electronics industry shall comply with the special requirements given in Table 3 when tested according to the methods prescribed in Annex B.

**3.3 Stannous Chloride****3.3.1 Description**

The material shall be in the form of colourless white crystals free from dirt, foreign matter and visible impurities. This is commercially known as tin salt.

**3.3.2 Composition**

The material shall correspond in composition essentially to  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ .

**3.3.3** The material shall also comply with the requirements given in Table 4, when tested according to the methods prescribed in Annex A and C.

**4 PACKING****4.1 Stannous Sulphate and Stannous Chloride**

Stannous sulphate and chloride shall be packed in

air-tight glass or earthenware containers, preferably with a replaceable closure. On keeping, the material rapidly hydrolyzes when exposed to air and hence shall not be packed in iron containers. It is, therefore, essential to store it always in air-tight containers to prevent deterioration of the material in storage.

**4.2 Sodium and Potassium Stannates**

Sodium and potassium stannates shall be packed in air-tight containers preferably with a replaceable closure. In the presence of moisture, the stannates rapidly deteriorate owing to the absorption of carbon dioxide and the salts pick up a tendency to become less soluble in water. Consequently, it is essential to store them at all times in air-tight containers.

**5 MARKING**

**5.1** The containers shall be marked with the name of the material, its net weight, identification of the source of manufacture and the date of manufacture.

**6 SAMPLING**

**6.1** The method of preparing representative test samples of the materials and the criteria for conformity of the material to the specification shall be as prescribed in Annex D.

**Table 2 Requirements for Sodium Stannate and Potassium Stannate**  
( Clause 3.2.3 )

Sl No.	Characteristic	Requirement for		Method of Test ( Ref to Cl No. of Annex B )
		Sodium Stannate	Potassium Stannate	
(1)	(2)	(3)	(4)	(5)
i)	Insoluble matter, percent by mass, <i>Max</i>	0·25	0·25	B-2
ii)	Total stannites ( as Sn ), percent by mass, <i>Max</i>	0·1	0·1	B-3
iii)	Soluble tin ( as Sn ), percent by mass, <i>Min</i>	40·0	38·0	B-4
iv)	Free alkali, percent by mass, <i>Max</i>	6·0 ( as NaOH )	1·5 ( as KOH )	B-5
v)	Lead ( as Pb ), percent by mass, <i>Max</i>	0·05	0·01	B-6
vi)	Arsenic ( as As ), percent by mass, <i>Max</i>	0·01	0·01	B-7
vii)	Nitrate, percent by mass, <i>Max</i>	0·25 ( as NaNO <sub>3</sub> )	0·25 ( as KNO <sub>3</sub> )	B-8
viii)	Chloride, percent by mass, <i>Max</i>	1·0 ( as NaCl )	1·0 ( as KCl )	B-9
ix)	Sodium ( as Na ), percent by mass, <i>Max</i>	—	0·01	B-10

**Table 3 Special Requirements for Potassium Stannate to be Used  
in Electronics Industry**  
( Clause 3.2.3.1 )

Sl No.	Characteristic	Requirement	Method of Test ( Ref to Cl No. of Annex B )
(1)	(2)	(3)	(4)
i)	Insoluble matter, percent by mass, <i>Max</i>	0·1	B-2
ii)	Free alkali ( as KOH ), percent by mass, <i>Max</i>	0·6	B-5
iii)	Lead ( as Pb ), percent by mass, <i>Max</i>	0·001	B-6
iv)	Nitrates ( as KNO <sub>3</sub> ), percent by mass, <i>Max</i>	0·1	B-8
v)	Chlorides ( as KCl ), percent by mass, <i>Max</i>	0·4	B-9

**Table 4 Requirements for Stannous Chloride**  
( Clause 3.3.3 )

Sl No.	Characteristic	Requirement	Methods of Test ( Ref to Cl No. of Annexes A and C )
(1)	(2)	(3)	(4)
i)	Insoluble matter, percent by mass, <i>Max</i>	0·10	A-2
ii)	Stannous tin ( as Sn ), percent by mass, <i>Min</i>	52·60	A-3
iii)	Sulphate ( as SO <sub>4</sub> ), percent by mass, <i>Max</i>	To pass the test	C-2
iv)	Alkalies and other metallic impurities ( as sulphates ), percent by mass, <i>Max</i>	0·10	C-3
v)	Arsenic ( as As ), percent by mass, <i>Max</i>	0·01	C-4

## ANNEX A

( Clauses 3.1.3 and 3.3.3 )

### ANALYSIS OF STANNOUS SULPHATE

#### A-1 QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals and distilled water ( see IS 1070 : 1977 ) shall be used in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

#### A-2 DETERMINATION OF INSOLUBLE MATTER IN STANNOUS SULPHATE

##### A-2.1 Reagents

###### A-2.1.1 Dilute Sulphuric Acid

2 percent ( v/v ) ( see IS 266 : 1976 ).

###### A-2.1.2 Dilute Sulphuric Acid

10 percent ( v/v ) ( see IS 266 : 1976 ).

##### A-2.2 Procedure

Weigh accurately about 10 g of the material and dissolve in 200 ml of dilute sulphuric acid ( 10 percent ). Filter through an 11 cm filter paper ( Whatman No. 40 is suitable ) containing a little filter pulp. Wash the residue with 100 ml of dilute sulphuric acid ( 2 percent ) and then wash free from acid with water. Transfer the filtrate and washing to a 500 ml graduated flask, make up to the mark with dilute sulphuric acid ( 2 percent ) and reserve ( solution A ). Ignite the residue in a silica crucible, cool and weigh.

##### A-2.3 Calculation

$$\text{Insoluble matter, percent by mass} = \frac{M_1}{M} \times 100$$

where

$M_1$  = mass in grams of the residue, and

$M$  = mass in grams of material taken for the test.

### A-3 DETERMINATION OF STANNOUS TIN

#### A-3.1 Reagents

##### A-3.1.1 Dilute Sulphuric Acid

2 percent (*v/v*).

##### A-3.1.2 Hydrochloric Acid

(see IS 265 : 1987).

##### A-3.1.3 Standard Potassium Iodate/Iodide

Dissolve 6.02 g of potassium iodate (previously dried at 105 to 110°C) in 400 ml of water containing 1 g of sodium hydroxide. Add 30 g of potassium iodide and dilute to 1 litre in a graduated flask. One millilitre of this solution is equivalent to 0.01 g of tin (as Sn).

##### A-3.1.4 Starch

Make a suspension of 0.5 g of starch in 10 ml of water. Add to it 90 ml of boiling water, cool and dilute to 100 ml. Prepare freshly each day.

#### A-3.2 Procedure

Dissolve 0.800 g of the material in 10 to 20 ml of dilute sulphuric acid. To the solution add 5 ml of hydrochloric acid, 20 ml of water and 5 ml of starch solution. Titrate with a minimum of shaking with standard potassium iodate/iodide solution to a permanent blue colour.

#### A-3.3 Calculation

$$\text{Stannous tin (as Sn), percent by mass} = V \times 1.25$$

where

$V$  = volume in millilitres of standard potassium iodate/iodide solution used in the test.

### A-4 DETERMINATION OF FREE SULPHURIC ACID

#### A-4.1 Reagents

##### A-4.1.1 Standard Sodium Hydroxide Solution

0.1 N approximately.

##### A-4.1.2 Phenolphthalein Indicator

Dissolve 0.1 g in 60 ml of ethanol and add 40 ml of water.

#### A-4.2 Procedure

Weigh accurately about 2 g of the material and shake for 2 minutes with 100 ml of ethanol which has previously been made just alkaline to phenolphthalein by careful addition of standard sodium

hydroxide solution. Allow to settle, decant 50 ml through a dry filter paper and titrate 50 ml of the filtrate with standard sodium hydroxide solution using phenolphthalein as indicator.

#### A-4.3 Calculation

$$\text{Free sulphuric acid (as H}_2\text{SO}_4\text{), percent by mass} = \frac{9.8 VN}{M}$$

where

$V$  = volume in millilitres of standard sodium hydroxide solution,

$N$  = normality of standard sodium hydroxide solution, and

$M$  = mass in grams of the material taken for the test.

### A-5 DETERMINATION OF CHLORIDE

#### A-5.1 Reagents

##### A-5.1.1 Concentrated Nitric Acid

(see IS 264 : 1976).

##### A-5.1.2 Standard Silver Nitrate Solution

0.1 N.

##### A-5.1.3 Standard Ammonium Thiocyanate Solution

0.1 N.

##### A-5.1.4 Ammonium Ferric Sulphate Solution

10 percent (*m/v*); dissolve 10 g of ammonium ferric sulphate in water, add 10 ml of nitric acid and dilute to 100 ml.

#### A-5.2 Procedure

To 100 ml of solution A (see A-2.2) add 10 ml of concentrated nitric acid and heat to oxidize the stannous tin. Cool, add 10 ml of standard silver nitrate solution and 5 ml of ammonium ferric sulphate solution and titrate the excess silver nitrate with standard ammonium thiocyanate solution to the appearance of a faint brown coloration.

#### A-5.3 Calculation

$$\text{Chloride (as Cl), percent by mass} = \frac{0.3546 (10 - V)}{M}$$

where

$V$  = volume in millilitres of standard ammonium thiocyanate solution required; and

$M$  = mass in grams of the material, equivalent to 100 ml of solution taken for the test.

### A-6 DETERMINATION OF ANTIMONY

#### A-6.1 Reagents

##### A-6.1.1 Concentrated Hydrochloric Acid

(see IS 265 : 1987).

**A-6.1.2 Standard Sodium Thiosulphate Solution**

0·01 N.

**A-6.1.3 Starch Solution**

( see A-3.1.4 ).

**A-6.2 Procedure**

Weigh accurately about 5 g of the sample and dissolve in 100 ml of water and 40 ml of concentrated hydrochloric acid. Add 1·5 g of potassium chlorate and boil ( for about 15 minutes ) to expel all traces of chlorine. Cool, dilute to 200 ml, add 1 g of potassium iodide and titrate the liberated iodine with standard sodium thiosulphate solution using starch as indicator.

**A-6.3 Calculation**

$$\text{Antimony ( as Sb ), percent by mass} = \frac{0\cdot06 V}{M}$$

where

$V$  = volume in millilitres of standard sodium thiosulphate solution used, and

$M$  = mass in grams of the material taken for the test.

NOTE — This method is not specific for antimony, but other metals liable to cause interference are not likely to be present in significant quantities. If present they shall be reported as antimony.

**A-7 DETERMINATION OF ARSENIC****A-7.1 Reagents****A-7.1.1 Dilute Hydrochloric Acid**

20 percent ( v/v ); ( see IS 265 : 1977 ).

**A-7.1.2 Standard Arsenic Solution**

Dissolve 0·132 g of arsenious oxide in 2 ml of 1 N sodium hydroxide solution, dilute to approximately 50 ml with water, add 1 ml of concentrated hydrochloric acid and make up the volume to 1 000 ml. Dilute 10 ml of this solution to 1 litre with water. One millilitre of this solution is equivalent to 0·001 mg of arsenic ( as As ).

**A-7.2 Procedure**

To 5 ml of solution A ( see A-2.2 ), add 20 ml of dilute hydrochloric acid and a few drops of stannous chloride solution and distil off 15 ml. To the distillate add 45 ml of water and test in accordance with IS 2088 : 1983, adding 10 g of arsenic free granulated zinc. Compare the stain produced on the mercuric chloride paper with a series of stains, produced in the same manner using known volumes of standard arsenic solution as follows:

To 50 ml of water in the Gutzeit apparatus add 10 ml of hydrochloric acid ( sp gr 1·16 to 1·18 ) and a few drops of stannous chloride solution and quantities from 1 to 10 ml of standard arsenic solution.

**A-7.3 Calculation**

$$\text{Arsenic ( as As ), percent by mass} = \frac{0\cdot0001 V}{M}$$

where

$V$  = volume in millilitre of standard arsenic solution required to produce matching stain; and

$M$  = mass in grams of the material, equivalent to 5 ml of solution taken for the test.

**ANNEX B**

( Clauses 3.2.3 and 3.2.3.1 )

**ANALYSIS OF SODIUM STANNATE AND POTASSIUM STANNATE****B-1 QUALITY OF REAGENTS**

**B-1.1** Unless specified otherwise, pure chemicals and distilled water ( see IS 1070 : 1977 ) shall be used in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

**B-2 DETERMINATION OF INSOLUBLE MATTER IN SODIUM AND POTASSIUM STANNATES****B-2.1 Reagents****B-2.1.1 Sodium Hydrochloric Solution**

1 percent ( m/v ).

**B-2.2 Procedure**

Weigh accurately about 25 g of the material and dissolve in 250 ml of sodium hydroxide solution at  $80 \pm 2^\circ\text{C}$ . After maintaining a temperature of  $80 \pm 2^\circ\text{C}$  for 30 minutes, filter the solution through an 11 cm filter paper ( Whatman No. 40 or equivalent ) containing a little filter pulp. Wash the residue with 100 ml of sodium hydroxide solution and then wash free from alkali with water. Transfer the filtrate and washings to a

500-ml graduated flask, make up to the mark with sodium hydroxide solution and reserve for subsequent tests ( solution B ). Ignite the residue in a silica crucible, cool and weigh.

### B-2.3 Calculation

$$\text{Insoluble matter, percent by mass} = \frac{M_1}{M} \times 100$$

where

$M_1$  = mass in grams of the residue, and

$M$  = mass in grams of the material taken for the test.

## B-3 DETERMINATION OF TIN ( TOTAL STANNITES )

### B-3.0 General

Tin is determined by titration with standard iodine solution.

### B-3.1 Reagents

#### B-3.1.1 Concentrated Hydrochloric Acid

( see IS 265 : 1984 ).

#### B-3.1.2 Standard Iodine Solution

0·1 N ( see IS 2316 : 1968 ).

#### B-3.1.3 Starch Solution

Triturate 5 g of starch and 0·01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into 1 litre of boiling water, boil for 3 minutes, allow the solution to cool and decant off the clear liquid.

### B-3.2 Procedure

Weigh accurately about 5 g of the material and dissolve in air-free distilled water. Acidify with 25 ml of hydrochloric acid and dilute to about 150 ml with air-free distilled water. Titrate immediately with standard iodine solution, using starch solution as indicator, until the blue colour produced persists.

### B-3.3 Calculation

$$\text{Total stannites ( as Sn ), percent by mass} = \frac{0\cdot5935 VN}{M}$$

where

$V$  = volume in millilitres of standard iodine solution used,

$N$  = normality of standard iodine solution, and

$M$  = mass in grams of the material taken for the test.

## B-4 DETERMINATION OF SOLUBLE TIN

### B-4.0 General

Soluble stannate is determined by titration with standard iodine solution.

### B-4.1 Apparatus

The apparatus consists of a 1 000 ml wide-necked conical flask with a 3-hole bung. One hole carries a tube connected to a source of carbon dioxide, the second to a tap funnel, while the third is used to insert a burette and is closed by a glass rod when not in use.

### B-4.2 Reagents

#### B-4.2.1 Concentrated Hydrochloric Acid

See IS 265 : 1987.

#### B-4.2.2 Nickel Coil

A suitable nickel coil is made from a piece of pure nickel sheet about 30 × 5 cm. A new coil should be etched before use by boiling for 10 minutes in a mixture of equal parts of hydrochloric acid ( 1 : 1 ) and sodium chloride solution ( 20 percent m/v ).

#### B-4.2.3 Carbon Dioxide Gas

#### B-4.2.4 Standard Iodine Solution

0·1 N ( see IS 2316 : 1968 ).

#### B-4.2.5 Starch Solution

See B-3.1.3.

### B-4.3 Procedure

Place 10 ml of the solution B reserved in B-2.2 in the flask and add 75 ml of concentrated hydrochloric acid and 200 ml of water. Place the nickel coil in the solution and pass a rapid current of carbon dioxide through the flask for a few minutes to expel all air. Reduce the gas flow, to a slow stream and boil the solution vigorously for 40 to 45 minutes. Increase the gas flow close the tap funnel and allow the flask to cool. When cold, add 2 ml of starch solution through the tap funnel, insert the burette and titrate with standard iodine solution to a permanent blue colour.

### B-4.4 Calculation

$$\text{Soluble tin ( as Sn ), percent by mass} = \frac{0\cdot5935 VN}{M}$$

where

$V$  = volume in millilitres of standard iodine solution used in the titration;

$N$  = normality of standard iodine solution; and

$M$  = mass in grams of the material, equivalent to 10 ml of solution taken for the test.

**B-5 DETERMINATION OF FREE ALKALI****B-5.0 General**

Free alkali is determined by titrating with standard hydrochloric acid solution.

**B-5.1 Reagents****B-5.1.1 Barium Chloride Solution**

10 percent (*m/v*).

**B-5.1.2 Thymolphthalein Indicator Solution**

Dissolve 0.1 g of thymolphthalein in 100 ml of rectified spirit (see IS 323 : 1959).

**B-5.1.3 Standard Hydrochloric Acid**

0.1 N.

**B-5.2 Procedure**

Weigh accurately about 2 g of the material and dissolve in 100 ml of freshly-boiled warm water. Allow to cool, add 50 ml of barium chloride solution and 1 drop of thymolphthalein indicator solution, and titrate with standard hydrochloric acid. Continue the titration with vigorous shaking until the blue colour is discharged. Add a further drop of indicator and continue titration. Take the end point when the solution changes from a definite blue to almost colourless.

**B-5.3 Calculation**

$$\text{Free alkali (as NaOH), percent by mass} = \frac{4VN}{M}$$

or

$$\text{Free alkali (as KOH), percent by mass} = \frac{5.6 VN}{M}$$

where

*V* = volume in millilitres of standard hydrochloric acid used in the titration,

*N* = normality of standard hydrochloric acid solution, and

*M* = mass in grams of the material taken for the test.

**B-6 DETERMINATION OF LEAD****B-6.0 General**

The colour produced by the addition of sodium sulphide in the solution is compared to that produced in the standard lead solution.

**B-6.1 Apparatus****B-6.1.1 Nessler Cylinders**

.50 ml capacity (see IS 4161 : 1967).

**B-6.2 Reagents****B-6.2.1 Sodium Hydroxide Solution**

(see B-2.1.1).

**B-6.2.2 Potassium Cyanide Solution**

10 percent (*m/v*).

**B-6.2.3 Sodium Sulphide Solution**

10 percent (*m/v*).

**B-6.2.4 Standard Lead Solution**

Dissolve 1.60 g of lead nitrate in water, add 1 ml of concentrated nitric acid (see IS 264 : 1976) and make up the volume to 1 000 ml. Transfer exactly 10 ml of the solution into a 1 000 ml volumetric flask and again dilute to the mark. One millilitre of the diluted solution contains 0.01 mg of lead (as Pb). The diluted solution should be freshly prepared.

**B-6.3 Procedure**

Take in a Nessler cylinder solution B, reserved in B-2.2, equivalent to 0.1 g of the material. Add 40 ml of sodium hydroxide solution, 2 ml of potassium cyanide solution and 10 drops of sodium sulphide solution and dilute with water up to 50 ml mark. Carry out a control test using 5 or 1 or 0.1 ml of standard lead solution in place of the solution of sodium stannate or potassium stannate or potassium stannate for electronic industry, as the case may be, along with the same quantities of other reagents as with the material, and diluting to 50 ml mark with water.

**B-6.3.1** The limits prescribed in Table 2 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

**B-7 DETERMINATION OF ARSENIC****B-7.0 General**

Arsenic is determined by modified Gutzeit method of test for arsenic.

**B-7.1 Reagents****B-7.1.1 Dilute Hydrochloric Acid**

20 percent (*v/v*). Dilute 20 ml of concentrated hydrochloric acid (see IS 265 : 1987) to 100 ml with water.

**B-7.1.2 Stannous Chloride Solution**

Dissolve 30 g of stannous chloride in 10 ml of hydrochloric acid and dilute to 100 ml with water.

**B-7.2 Procedure**

Take solution B, reserved in B-2.2, equivalent to 0.1 g of the material. Add 20 ml of dilute hydrochloric acid and a few drops of stannous chloride

solution and distil off 15 ml. To the distillate add 45 ml of water and carry out the test for arsenic as prescribed in IS 2088 : 1983.

**B-7.2.1** The limit prescribed in Table 2 shall be taken as not having been exceeded if the stain produced is not greater than that produced by 0·01 mg of arsenic.

## B-8 DETERMINATION OF NITRATES

### B-8.0 General

Nitrates are reduced to ammonia, which is observed in standard acid and excess of acid is titrated with standard alkali solution.

### B-8.1 Apparatus

The apparatus, as assembled, is shown in Fig. 1. It consists of a round bottom flask *A* of 1 000 ml capacity fitted with a rubber stopper through which passes one end of the connecting bulb tube *B*. The other end of the bulb tube *B* is connected to the condenser *C* by a rubber stopper and the lower end of the condenser *C* is attached by means of a rubber tubing to a dip tube *D* which dips into beaker *E* of 250 ml capacity.

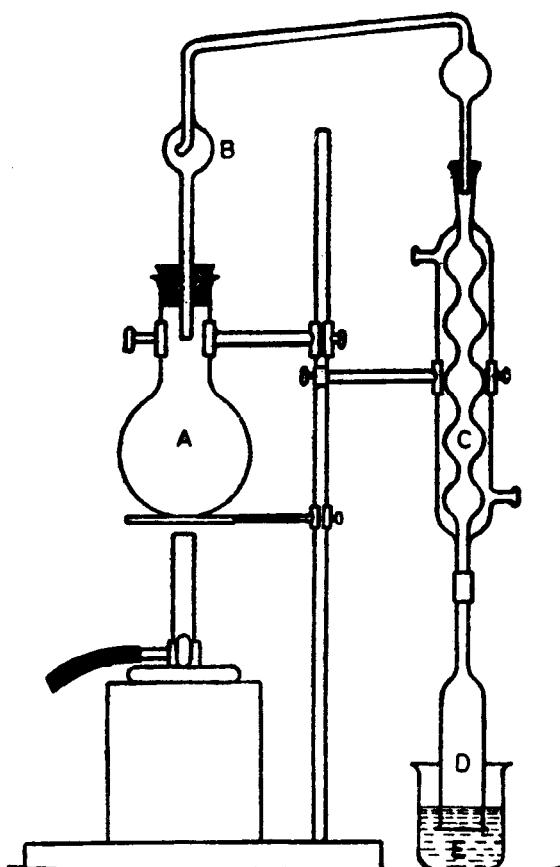


FIG. 1 APPARATUS FOR THE DETERMINATION OF NITRATES

### B-8.2 Reagents

#### B-8.2.1 Sodium Hydroxide

0·1 N approximately.

#### B-8.2.3 Devarda's Alloy

#### B-8.2.4 Standard Sodium Hydroxide Solution

0·1 N approximately.

#### B-8.2.5 Methyl Red Indicator Solution

Dissolve 0·15 g of methyl red in 500 ml of water.

### B-8.3 Procedure

Take 200 ml of solution, reserved in **B-2.2**, in the flask *A*, add 20 g of sodium hydroxide and 2·5 g of Devarda's Alloy and assemble the apparatus as shown in Fig. 1. Take 25 ml of standard hydrochloric acid in beaker *E*; and gently warm the flask *A*. When the evolution of gas has ceased, boil the contents of the flask vigorously for 15 minutes. Titrate the solution in the beaker with standard sodium hydroxide solution using methyl red as indicator. Carry out a blank determination as above.

### B-8.4 Calculation

$$\text{Nitrates (as NaNO}_3\text{), percent by mass} = \frac{8.501 (V - V_1) N}{M}$$

or

$$\text{Nitrates (as KNO}_3\text{), percent by mass} = \frac{10.11 (V - V_1) N}{M}$$

where

*V* = volume in millilitres of standard sodium hydroxide solution used in titration in the blank determination;

*V*<sub>1</sub> = volume in millilitres of standard sodium hydroxide solution used in titration with the material;

*N* = normality of standard sodium hydroxide solution; and

*M* = mass in grams of the material, equivalent to 200 ml of solution *B* (see **B-2.2**) taken for the test.

## B-9 DETERMINATION OF CHLORIDES

### B-9.1 Reagents

#### B-9.1.1 Dilute Nitric Acid

(1 : 4).

#### B-9.1.2 Standard Silver Nitrate Solution

0·1 N (see IS 2316 : 1968).

**B-9.1.3 Nitrobenzene****B-9.1.4 Standard Ammonium Thiocyanate Solution**

0·1 N (*see IS 2316 : 1968*).

**B-9.1.5 Ferric Ammonium Sulphate Indicator Solution**  
saturated.**B-9.2 Procedure**

Acidify 100 ml of the solution *B* reserved in **B-2.2** with dilute nitric acid and dilute to 250 ml with water. Add 25 ml of standard silver nitrate solution and 10 ml of nitrobenzene and shake well. Titrate the excess of silver nitrate with standard ammonium thiocyanate solution using 5 ml ferric ammonium sulphate solution as indicator.

**B-9.3 Calculation**

$$\text{Chlorides ( as NaCl ), percent by mass} = \frac{0\cdot5845(25 - V)}{M}$$

or

$$\text{Chlorides ( as KCl ), percent by mass} = \frac{0\cdot7456(25 - V)}{M}$$

where

*V* = volume in millilitres of standard ammonium thiocyanate solution used in titration; and

*M* = mass in grams of the material, equivalent to 100 ml of solution *B* (*see B-2.2*) taken for the test.

**B-10 DETERMINATION OF SODIUM**

**B-10.1** Determine sodium with a flame photometer in accordance with the test method prescribed in IS 9497 : 1980.

## **ANNEX C**

*( Clause 3.33 )*

**ANALYSIS OF STANNOUS CHLORIDE****C-1 QUALITY OF REAGENTS**

**C-1.1** Unless specified otherwise, pure chemicals and distilled water (*see 1070 : 1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

**C-2 DETERMINATION OF SULPHATES****C-2.1 Reagents****C-2.1.1 Dilute Hydrochloric Acid**

approximately 5 N.

**C-2.1.2 Barium Chloride Solution**

prepared by dissolving about 12·2 g of barium chloride in 100 ml of water.

**C-2.2 Procedure**

Weigh accurately 2 g of the material and dissolve in 50 ml of water and 1 ml of dilute hydrochloric acid. Add 1 ml of barium chloride solution and allow to stand for 1 hour.

**C-2.2.1** The material shall be taken to have satisfied the requirement of the test if no turbidity or precipitate is produced.

**C-3 DETERMINATION OF ALKALIS AND OTHER METALLIC IMPURITIES****C-3.1 Reagents****C-3.1.1 Dilute Hydrochloric Acid**

10 percent (*v/v*).

**C-3.1.2 Sulphuric Acid**

(*see IS 266 : 1977*).

**C-3.2 Procedure**

Weigh accurately about 2 g of the material and dissolve in 100 ml of dilute hydrochloric acid. Pass hydrogen sulphide through the solution gently till all the tin salts are precipitated. Filter through a filter paper (Whatman No. 40 is suitable) and evaporate carefully over low heat to dryness. Cool and add a drop of sulphuric acid to the residue and ignite gently. When the fumes cease, cool the residue in a desiccator and weigh.

**C-3.3** The limit prescribed in Table 3 shall be taken as not having been exceeded, if the residue does not weigh more than 2 mg.

**C-4 DETERMINATION OF ARSENIC****C-4.1 Reagents****C-4.1.1 Hydrochloric Acid**

20 percent (*v/v*).

**C-4.2 Procedure**

Weigh accurately about 5 g of the material. Dissolve it in 20 ml of hydrochloric acid. Transfer the solution to a distillation flask and distil carefully. Collect the first 15 ml of the distillate and

add to it 45 ml of water. Test for arsenic as prescribed in IS 2088 : 1962.

**C-4.3** The limit prescribed in Table 3 shall be taken as not having been exceeded if the stain produced is not greater than that produced by 0.5 mg of arsenic.

**ANNEX D**

( Clause 6 )

**SAMPLING OF TIN SALTS FOR ELECTROPLATING****D-1 GENERAL REQUIREMENTS FOR SAMPLING**

**D-1.1** Samples shall be taken in a protected place not exposed to damp air, dust or soot.

**D-1.2** The sampling instrument shall be clean and dry.

**D-1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**D-1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**D-1.5** The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

**D-1.6** The sample containers shall be of such a size that they are almost completely filled by the sample.

**D-1.7** Each sample container shall be sealed airtight after filling, and marked with full details of sampling, the date of sampling and details given under 5.

**D-2 SCALE OF SAMPLING****D-2.1 Lot**

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

**D-2.2** For ascertaining the conformity of the material to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected at random

from lots of different sizes shall be as given below:

<i>Lot Size ( N )</i>	<i>Sample Size ( n )</i>
3 to 15	3
16 " 40	4
41 " 110	5
111 " 180	6
181 " 300	7
301 " 500	8
501 " 800	9
801 and above	10

**D-2.3** In order to ensure randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure is recommended for use:

Starting from any one count all the containers in the lot in one order as 1, 2, ...., up to  $r$  and so on, where  $r$  is the integral part of  $N/n$  ( see D-2.2 ). Every  $r$ th container thus counted shall be withdrawn to constitute the sample.

**D-3 PREPARATION OF TEST SAMPLES**

**D-3.1** From each of the containers selected in D-2.3 small portions of material shall be taken from different parts with the help of a suitable sampling instrument. These portions when mixed together shall constitute the individual sample representing the container. The material in each individual sample left after preparation of the composite sample as prescribed in D-3.2 shall be not less than 3 times the material required for testing as prescribed in D-4.1, and shall be divided into 3 equal parts one each for the buyer and the supplier and the third as referee sample.

**D-3.2** From all the individual samples obtained in D-3.1, small but equal quantities of material shall be taken and mixed to constitute the composite sample. The material in the composite sample shall be at least 3 times the material required for testing prescribed in D-4.2, and shall be divided into 3 equal parts one each for the buyer and the supplier and the third as referee sample.

**D-3.3** All the samples obtained after division

in **D-3.1** and **D-3.2** shall be transferred to separate sample containers (*see D-1.5 and D-1.6*) which shall be closed air-tight, sealed and marked with full identification particulars (*see D-1.7*).

**D-3.4** The set of referee samples, consisting ( like the other two sets ) of a composite sample and individual samples, shall bear the seal of both the purchaser and the supplier, and shall be kept under conditions suitable to preserve the characteristics of the material.

#### **D-4 NUMBER OF TESTS**

**D-4.1** Each of the individual samples shall be separately tested for the following requirements:

- a) Stannous tin and free sulphuric acid in the case of stannous sulphate,
- b) Total stannites and free alkali in the case of sodium and potassium stannates, and
- c) Stannous tin and sulphate in the case of stannous chloride.

**D-4.2** The composite sample shall be tested for all the requirements not tested on the individual samples.

#### **D-5 CRITERIA FOR CONFORMITY**

##### **D-5.1 For Individual Samples**

For those characteristics which are on individual

samples, the mean and the range of test results shall be computed as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{sum of individual test results}}{\text{number of tests}}$$

$$\text{Range } (R) = \text{difference between the maximum and the minimum test results}$$

**D-5.1.1** The lot shall be declared to have satisfied the requirements for the characteristics tested if the corresponding criteria given below are fulfilled:

<i>Characteristics Specified as</i>	<i>Criteria for Conformity</i>
a) Minimum	$\bar{X} - 0.6 R \geqslant \text{minimum specified}$
b) Maximum	$\bar{X} + 0.6 R \leqslant \text{maximum specified}$

##### **D-5.2 For Composite Sample**

For declaring the conformity of the lot to the requirements of all other characteristics, the test results on the composite sample shall meet the corresponding requirements specified.

#### **Standard Mark**

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

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### Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002  
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksantha  
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### Regional Offices :

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg  
NEW DELHI 110002

Telephone

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331 13 75

Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Manikula  
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87 86 62

Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036

53 38 43

Southern : C. I. T. Campus, IV Cross Road, MADRAS 600113

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